HIGH TEMPERATURE APPLICATIONS OF STRUCTURAL CERAMICS

QUARTERLY PROGRESS REPORT

July - September 1981

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U. S. Department of Commerce
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PREPARED FOR THE UNITED STATES
DEPARTMENT OF ENERGY

Under Contract No. DE-A105-800R20679

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INTRODUCTION

The achievement of higher efficiency thermochemical engines and heat recovery systems requires the availability of high temperature, high performance structural materials. Structural ceramics such as SiC, Si_3N_4 and certain Al_2O_3-Si_3N_4 combinations have received particular attention for these applications due to their basic characteristics of good strengths coupled with good corrosion and thermal shock resistances. Even with these positive attributes, improved reliabilities and extended lifetimes under service conditions are necessary for structural ceramics to gain industrial acceptance and use. The problems are mechanical and/or chemical in nature and are enhanced by the fact that these materials are subjected to high temperatures, reactive environments and extreme thermal gradients.

With an objective of improved performance for heat engine/heat recovery applications the NBS program on structural ceramics addresses these problems through the determination of the critical factors which influence mechanical and microstructural behavior. The activities of the program are grouped under four major subtasks with each designed to develop key data, associated test methods and companion predictive models. The status of the subtasks are detailed in the following sections.

NBS 5.2.10(A) - HIGH TEMPERATURE FRACTURE OF STRUCTURAL CERAMICS

RESULTS FROM PRIOR QUARTERS

Four-point bend tests on notched bars of Si_5AlON_7 were completed and the results compared with previous work on an yttria-doped SiC (NCX34) and with literature values for Si_3N_4. Reduced scale testing rigs using SiC were manufactured to test small billets of experimental materials. Fifteen different compositions of Si_3N_4 + Y.A.G. materials obtained from Professor Tien of University of Michigan using these rigs were tested at 1300 °C in order to avoid oxidation. Further studies of Tien's Si_3N_4 were conducted to ascertain critical stress intensity factors and to clarify some inconsistencies in earlier results. A literature survey of available data on fracture toughness, crack growth behavior and creep properties of SiC, Si_3N_4 and sialon was completed. Four-point bending tests under constant displacement rate were performed on several SiC and sialon specimens. For α-SiC tested in air, the results showed little oxidation and high resistance to slow crack growth up to 1300 °C. For sialon specimens ($Si_{6-z}Al_zO_zN_{8-z}$), it was found that as z increases, the susceptibility to crack growth and oxidation decreases.

The asymptotic behavior of the stress intensity factor as a function of crack length for 4-point bending at small and large crack lengths was investigated in more detail. This work is useful for future numerical solution schemes. Also, an analysis of load-deflection curves which compute crack growth behavior (V-K) without requiring a knowledge of Young's modulus or machine compliance was completed.

High temperature four-point bending tests under constant displacement rate condition have been performed on <u>notched</u> reaction sintered SiC specimens. Several ways of initiating cracks were tried before a chevron notch was utilized. This produced a stable crack growth period and a sharp crack.

A paper entitled "Slow Crack Growth in Sialon" was presented at the International Symposium on Fracture Mechanics of Ceramics, University Park, Pennsylvania July 17, 1981. A paper entitled "Effects of Deformation on the Fracture Behavior of Si_3N_4 and Sialons" was presented at the 2nd NATO

Advanced Study Institute held at Falmer, England from the 27th of July to the 7th of August, 1981. Both of these papers presented work which came partly from this task.

DISCUSSION OF CURRENT ACTIVITIES

Further analyses of the four-point bend test were completed. One analysis allows the determination of the tensile creep behavior from flexural testing of trapezoidal cross-sections. A further analysis deducing tensile behavior from bending and compression tests will be finished shortly. These results will be written up and published. A sensitivity analysis just performed on four-point bending allows us to judge what type of crack growth behavior we may be able to measure properly in flexure. Materials not having these crack growth characteristics require a mechanically more stable test specimen such as the double-torsion specimen. This has proved to be the case for α-SiC and we are now fabricating these sorts of specimens from this material. We are still awaiting the delivery of β-SiC to complete work on this material. In the meanwhile, we are working ahead on the properties of the siliconized silicon carbide.

NBS 5.2.10(B) - CRACK GROWTH MECHANISM MAPS

RESULTS FROM PRIOR QUARTERS

Preliminary maps for Si_3N_4 have been drawn. A computer program for automatically drawing maps from K-V data has been written. Data for SiC has been collected with the aim of constructing a crack growth mechanism map. An evaluation of K_{IC} was made because its definition by ASTM E-399 conflicts with common usage in the ceramic community. A preliminary crack growth mechanism map for a commercial hot pressed SiC was constructed from published information. The literature survey of fracture and crack growth mechanisms was completed for SiC, Si_3N_4 and sialon.

A crack growth model based on diffusion mechanisms was developed. The model entails a grain-boundary crack growing in steady state due to applied stress by stress assisted surface and grain-boundary diffusion. By further assuming the grain on either side of the crack behaves elastically, the model predicts a unique K-V relationship and a threshold K below which no crack growth will take place. A comparison of this prediction to a set of creep crack growth data on sialon at 1400 °C showed good agreement.

The paper entitled "A diffusive crack growth model for creep fracture" (NBSIR 81-2255) was accepted for publication in J. Am. Ceram. Soc.

An investigation into the energy release rate of this crack growth model was included in this paper. The results show that J is the total amount of energy consumption rate which consists of a portion consumed in the creation of new crack surfaces and the remaining portion dissipated in the matter transport processes.

The theoretical prediction of V-K functional relationship based on diffusion-controlled crack growth was mapped into K-T space in order for the future data to fit in the diffusive crack growth area of the mechanism maps.

A paper entitled, "Crack Growth Mechanism Maps" was published in Advances in Fracture Research. This paper describes the concept of crack growth mechanism maps and details their construction.

DISCUSSION OF CURRENT ACTIVITIES

Data obtained in part A above is being analyzed here in order to construct a crack growth mechanism map for NCX34. Since this material (i.e., Y_2O_3 doped silicon carbide) is one of the most promising nitrogen ceramics, the map may prove very useful.

Due to the work performed in this DoE sponsored research project, the investigators were invited to participate in a recent NATO Advanced Study Institute on nitrogen ceramics. Also, we have been invited to present our work on time dependent failure mechanisms in silicon carbide to next summer's Gordon Conference on ceramics. Much of the work to be presented directly emanates from this DoE project. Our main emphasis in the review

and evaluation of crack growth theories has been in diffusive crack growth theories. We believe that this is the long term mechanism of failure and, hence, the one of technological importance.

NBS-5.2.10(C) - MICROSTRUCTURE AND PHASE ALTERATION

RESULTS FROM PRIOR QUARTERS

X-ray analyses were performed on various sialon and as-received silicon carbide samples. Also, preliminary x-ray data were obtained on alpha silicon carbide oxidized in the temperature range from 1200 to 1500 °C.

A literature survey was begun to collect references on the oxidation and the high-temperature thermal and mechanical properties of silicon carbide.

A concept for a high-temperature x-ray furnace was developed in which the sample serves as the heating element.

DISCUSSION OF CURRENT ACTIVITIES

Phase Analysis

The various SiC polytypes, including β-SiC, can be indexed on hexagonal unit cells which differ principally in the stacking in the c-axis direction. In considering the structural polytypes of SiC, it is convenient to focus on the placement of Si-atoms, since the C-tetrahedra can be referenced with respect to the Si-sublattice. The Si-atoms can assume either a cubic packing or a hexagonal packing in the c-axis direction. The SiC polytypes are a consequence of various combinations of these two types of packing [1].

In view of the number of potential structures, it appears advisable to use a structural analysis procedure which takes account of these structural variations in a fundamental manner. Such a procedure is one that assigns occupation probabilities to each lattice site in the (001) planes above the first two fiducial planes for which the site occupations are

defined, a priori. This procedure recognizes that the x-rays inherently average over many unit cells so that occupation probabilities are consistent with the method of measurement. The resulting theoretically calculated structure factors and predicted diffraction pattern will have diffraction maxima which correspond to the range of structural polytypes, but each maxima will have a weight, or composite probability, assigned to it. Among peaks characteristic of a given polytype, relationships between the composite probabilities are to be expected. Comparison of observed peaks and peak intensities with the predicted pattern will establish the set of polytypes which are consistent with the data and will indicate those peaks which are assignable to impurities. It is also expected that this analysis can be employed in oxidation studies to target groups of peaks for observation during the oxidation process. Such an approach may identify those polytypes which are most subject to oxidation.

Theoretical calculation of the structural factors and predicted x-ray diffraction pattern are now in progress.

Structural Ceramics Bibliography

A structural ceramics bibliography is attached. Although SiC and oxidation studies were emphasized in the literature search, references also were collected for Si_3N_4 and Si_3N_4-oxide systems and for other properties.

High-Temperature X-ray Diffraction

Designs for a high-temperature x-ray diffraction furnace were prepared. These include drawings for the furnace housing (cover and endplate) and the ceramic insulation.

Oxidation Studies

SiC sample holders and setter plates were prepared and conditioned at 1500 °C. Currently, two furnaces for this study are being relocated to another laboratory, and work on this study is suspended until suitable

electrical power is installed.

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NBS-5.2.10(D) - MICROSTRUCTURE AND FRACTURE IN REACTIVE ENVIRONMENTS

RESULTS FROM PRIOR QUARTERS

An apparatus for mechanical testing structural ceramics in gaseous combustion products at temperatures up to 1400 °C has been designed, and the components required to construct the apparatus have been ordered from commercial suppliers. The apparatus includes a laboratory box furnace which is gas tight, a gas blending system which can control the flow and composition of a mixture of 5 gases (SO_2, H_2O, CO_2, O_2, and N_2), and an existing, displacement rate controlled, loading machine. The furnace will provide a 6 inch cube cavity that is heated electrically by molybdenum disilicide elements. The gas mixture at nominally one atmosphere pressure will flow through the furnace cavity. The gas blending system controls the flow of each gas component by the thermal conductivity of its mass.

DISCUSSION OF CURRENT ACTIVITIES

The furnace system is under construction by its manufacturer. We have conferred with the manufacturer to have the furnace modified so that load bearing rods can be introduced through top and bottom ports of the furnace. Regular access to the furnace cavity for placement of a test specimen between the bearing rods is made through a sealed, water-cooled front door.

Gas tight fixtures were designed for attachment to the top and bottom furnace posts so that loading could be transferred from the test machine to a specimen in the furnace. Commercial flanges and bellows required for the fixtures were obtained. Accessory components are presently being machined to hold the bearing rods. All of these parts will then be welded together to make the fixtures.

The commercial gas blending system was delivered. The system includes separate controllers which maintain the flow of each gas individually over a specified flow range before blending. Unfortunately, the supplier inadvertently transposed the ranges requested for SO_2 and N_2. The units for these two gases were returned for recalibration.

Alteration of the laboratory room facilities were initiated to provide power and cooling-water to the furnace and to exhaust the flow of gas from the furnace out of the room.

Although construction of the apparatus is not yet completed, we expect that its assembly will soon be underway and its operation will be verified on schedule by December 1981.